



16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Initial Calibration Curve (ICAL):

- 16.1.1 Frequency: A new curve must be generated when the ICV or CCV criteria are not met, or after major instrument maintenance such as column replacement or changes in operating conditions
- 16.1.2 Acceptance Criteria:
 - 16.1.2.1 ICAL curve must have 5-points minimally for all analytes;
 - 16.1.2.2 Linearity demonstrated when the mean CF RSD for all analytes must be $\leq 20\%$ or perform the linear regression and generate $r > 0.995$ for analytes where RSD is $> 20\%$.
 - 16.1.2.3 If non-linear fit (quadratic) is used, the $r^2 > 0.99$ and six calibration points are required.
- 16.1.3 Curve Failure Corrective Action:
 - 16.1.3.1 Check standards and/or perform maintenance as necessary to correct problem, then generate new curve.

16.2 Initial Calibration Verification (ICV):

- 16.2.1 Frequency: A new curve must be generated when the ICV or CCV criteria are not met, or after major instrument maintenance such as column replacement or changes in operating conditions.
- 16.2.2 Acceptance criteria: agreement between the curve and the ICV results must be between 80 - 120 % of the ICV true values on both columns.
- 16.2.3 ICV Failure Corrective action: evaluate condition and age of standards being used and/or perform any needed system maintenance. Do not analyze samples until the criteria can be met or prepare new standards and/or generate new curve if criteria cannot be met.

16.3 Absolute Retention Time (RT) and RT window:

- 16.3.1 Acceptance Criteria: All analytes and surrogates in ICV & CCV must fall within the RT windows
- 16.3.2 Frequency: Reset absolute RT with each new ICAL (use a midpoint standard in the new curve) and update absolute RT with each 12 shift CCV; and set 0.05 RT windows around the daily CCV absolute RT and verify as being ± 3 times standard deviation for each analyte retention time from 72-hour study. The ICV and CCVs must fall within the RT windows.
- 16.3.3 Absolute RT position Corrective action: If absolute RT falls outside criteria (0.05-min, as per section 11.16.10, perform maintenance to stabilize RT windows to fall within criteria.

16.4 Continuing Calibration Verification (CCV):

- 16.4.1 Frequency: The calibration standard (CCV) must be run at the beginning of each daily sequence, minimally after 10 samples (including QC samples) and at the end of the analytical sequence.
- 16.4.2 Acceptance Criteria: All analytes are $\pm 20\%$ of the expected values on both columns. The grand mean cannot be employed (SW 8000C requirement).
- 16.4.2 CCV failure Corrective Action: If the calibration does not meet the criteria, correct the problem and re-analyze the standard. If the CCV still fails, prepare as new calibration curve. All samples run since last passing CCV must be re-run.

16.5 Method Blank:

- 16.5.1 Frequency: Analyze the method blank at a frequency of one per preparation batch of 20 or less samples (it is prepared in that frequency).



The method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system is free of contaminants. If the method blank indicates contamination, it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

- 16.5.1.1 Acceptance Criteria: All analytes of interest must be less than the $\frac{1}{2}$ MQL; or Other approved QA program requirements must be followed when the acceptable blank contamination specified in the approved quality assurance project plan differs from the above.
- 16.5.1.2 analytes of interest must be less than 5% of the regulatory limit associated with an analyte or analytes of interest must be less than 5% of the sample result for the same analyte, whichever is greater.
- 16.5.2 Method Blank Contamination Corrective Action: If the method blank results do not meet the criteria above, then the laboratory must locate and reduce the source of the contamination and reanalyze any samples associated with the contaminated method blank that have detections of the affected analyte. If samples cannot be re-run because of insufficient sample or other similar circumstances, a corrective action report must be initiated and issued to project management and to the QAD. The NCAR must be detailed enough for preparation of the project narrative and all appropriate data flags are entered into LIMS for the final report preparation. Data reported with an associated contaminated method blank must be flagged with a "B", indicating the occurrence.
- 16.6 Laboratory Control Sample:
 - 16.6.1 LCS Frequency: The laboratory control sample is processed with each batch of 20 or less samples. All samples in the batch must be processed on the same day.
 - 16.6.2 Acceptance Criteria: refer to Table 21.2 for LCS recovery criteria.
 - 16.6.3 LCS failure Corrective Action: If the LCS recovery for the compounds of interest does not meet with the criteria, the sample batch must be re-analyzed. If reprocessing it is not possible due to lack of sample or expired hold time, report (narrate) the variance to the client and flag the associated data as estimated.
- 16.7 Matrix Spike and Matrix Spike Duplicate: Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate should be based on knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, use a matrix spike and matrix spike duplicate pair. If samples are expected to contain target analytes, then use one matrix spike and a duplicate analysis of an unspiked field sample is acceptable.
 - 16.7.1 Frequency: Perform MS and MSD at a frequency of at least one MS and MSD per 20 samples. If an MSD cannot be processed due to insufficient sample, a duplicate LCS shall be processed for the purpose of assessing duplicate RPD criteria.
 - 16.7.2 Acceptance Criteria: Spike recovery and RPD criteria are specified in the Table 21.2.
 - 16.7.3 MS/MSD % Recovery Criteria Failure Corrective Action: If the MS and MSD



have recoveries which are outside the target range, the poor recoveries in the MS and MSD may be due to matrix effects. The LCS, surrogate recoveries and calibration results must all be evaluated in order to determine if matrix interference is present or if method performance is poor. Note that the MS and MSD are used to evaluate the matrix effect, not to control the analytical process. If both the Matrix Spike and Matrix Spike Duplicate are found to be out of control for the same analyte, a matrix effect is likely confirmed (assuming the LCS passes). If recoveries of the MS/MSD pair are suspicious (laboratory error, etc) another set of MS/MSD must be re-extracted with the sample batch. For instance, if both matrix spikes exhibit low recovery but good precision then it can be assumed that matrix interference is present. However, if precision between the MS and the MSD is poor, technique error must be eliminated as a possible source of error before the data can be accepted. If matrix interference is highly suspected corrective action is not necessary.

- 16.7.4 MS/MSD RPD Criteria Failure Corrective Action: If the RPD fails, the data must be evaluated for error and perform sample/batch QC reprocessing if necessary.

16.8 Surrogates:

- 16.8.1 Frequency: Surrogates are spiked into each sample and QC sample. The recoveries must be evaluated for all samples and QC samples.
- 16.8.2 Criteria: Acceptance limits for each matrix are found in the Table 21.2.
- 16.8.3 Surrogate Failure Corrective Action: If the surrogate recoveries do not meet the criteria above check for possible errors in the calculations or surrogate solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re-extract and reanalyze sample, if possible. If obvious matrix effect is present, re-extraction may not be necessary. If upon re-analysis the recovery is outside limits, report the data describe in laboratory checklist and suspected matrix interference. Also, Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses should be reported. If no instrument problem is found, the sample should be re-extracted and re-analyzed. If, upon re-analysis, the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. If the holding time for the re-extraction has expired, check with project management whether reporting of both the original and re-analysis results are to be required and describe in the checklist.

- 16.9 Demonstration of Proficiency: Each analyst must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating 4 sets of data of acceptable accuracy and precision for target analytes in a



clean matrix. Each analyst must demonstrate ongoing proficiency annually with each sample preparation and determinative method combination it utilizes, by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix or by passing performance in approved PT evaluations. If analyst does not perform continuing DOC within a calendar year, then analyst must perform new initial demonstration prior to sample analysis.

- 16.10 Limits of Detection (LOD) must be evaluated as per SOP HS-QS-006 (LOD and LOQ).
- 16.11 Use the Lab Review checklist to describe anomalies. Mandatory QC failures (LCS, etc.) are documented using an NCAR form and submitted to the department supervisor and to the QA Manager.

17) Data Records Management

- 17.1 All data is stored both electronically and hard copy for 10 years.
- 17.2 All analytical sequence IDs and associated standards such as CCCVs must be recorded in the Runlogs.
- 17.3 Standard preparation such as ical standards, daily CCV, solvent, etc. must be recorded in the standard prep logbook. To simplify standard traceability, an effort must be made to use single lots of reagent and standards. Hardcopy computer printouts of analytical sequences and raw data must be retained and initialed by the analyst (electronic initials are acceptable). To simplify standard traceability, an effort must be made to use single lots of reagent and standards
- 17.4 Complete all pertinent sections in the respective logbooks. If not-applicable then line out the section. "Z" out or "X" out all large sections of the worksheet that are not used. Make all corrections with single line through, date and initial. Make NO obliterations when manually recording data.
- 17.5 Logbooks are controlled. Never remove a page from a logbook. Completed logbooks are returned to the QA department when filled and no longer needed in the work area.
- 17.6 SOP effective date is the date noted in the header or last signature date, whichever is most recent.
- 17.7 All maintenance must be documented in maintenance logbook documenting the service performed. Information such as instrument symptoms, serviced performed, parts changed including serial numbers and the return to service status, see Preventative Maintenance SOP.

18) Contingencies for Handling Out of Control Data

- 18.1 When method required QC exceedances occur, in every case where sample data quality are affected, the source of the QC exceedance must be determined, corrected and sample reanalysis carried out when possible.
- 18.2 When affected sample analysis can not be repeated due to limitations (i.e. sample availability, or if reanalysis can only be performed after expiration of a sample hold time), the reporting of data associated with exceeded QC data must be appropriately flagged and narrated. This documentation is necessary to define for the data user the effect of the error has upon the data quality of the results reported (e.g. E flag data indicate the result to be only an estimate).



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- 18.3 All analysts must report sufficient comments in laboratory data review checklist for exceeded QC associated with sample results so that project management can further narrate and ensure data qualifiers (flags) are properly assigned to the reported data.
- 18.4 NCARs must be issued for QC system exceedances. Matrix interferences are reported using the analyte reporting comment section in LIMS or using the Laboratory Data review checklist.

19) Method Performance

19.1 Refer to Section 9 of Reference 21.1.1.

20) Summary of Changes

Table 20.1 Summary of Changes

Revision Number	Effective Date	Document Editor	Description of Changes
08.1	07/01/2016	T. Yen	New Lab Director and Department Supervisor
08.1 – Section 4.7	07/01/2016	T. Yen	Job safety assessment performed for Method 8151A.
08.1 – Section 12	07/01/2016	T. Yen	Procedures updated
08.1 – Section 13	07/01/2016	T. Yen	Troubleshooting update – new calibration with column changes and MDL with major repairs.
08.1 – Section 15.8.1	07/01/2016	T. Yen	Moisture correction formula added.
08.1 – Section 16.10	07/01/2016	T. Yen	IDOC criteria added.
08.1 – Section 17.0	07/01/2016	T. Yen	Daily and calibration standards documentation and maintenance record documentations.
08.1 – Section 21.0	07/01/2016	T. Yen	Reference update.
08.0	09/08/2014	T. Yen	SOP Format Change and New Lab Director, Department Supervisor and QA Manager.
08.0 – Section 10.1	09/08/2014	T. Yen	Link standards/reagents to run/prep batches.
08.0 – Section 17.2	09/08/2014	T. Yen	Use of single lots of standard/reagent to simplify standard tracking.
08.0 – Section 17.5	09/08/2014	T. Yen	SOP effective date defined.
08.0 – Section 21	09/08/2014	T. Yen	Reference updated.
08.0 – Section 21	09/08/2014	T. Yen	Most current MDL reference to LIMS.
07.2	08/15/2012	J. Cady	Minor revision. Format/font change.
07.1	09/30/2011	J. Cady	Inserted Tables 10.6.2 and 10.6.3
07.0	08/01/2011	J. Cady	Major document revision.



21) References and Related Documents

21.1 Referenced Documents

- 21.1.1 U.S. Environmental Protection Agency, "Method 8151A "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.
- 21.1.2 U.S. Environmental Protection Agency, "Method 8000D, Determinative Chromatographic Separations", Update V, Revision 4, July 2014.
- 21.1.3 TNI 2009 Standards, TNI Institute, 2009.
- 21.1.4 Department of Defense Quality Systems Manual, Version 5, July 2013.

22) Appendix

Table 22.1 SW 8151A MQLs at time of SOP revision

Table 22.1 - SW 8151A MQLs, current at time of SOP revision. MDLs are determined annually at a minimum, and are subject to change. For most current MDL consult QA of LIMS.			
Analyte - water matrix	MQL µg/L	Analyte - solid/soil matrix	MQL µg/Kg
2,4,5-T	0.1	2,4,5-T	3.3
2,4,5-TP (Silvex)	0.1	2,4,5-TP (Silvex)	3.3
2,4-D	0.2	2,4-D	6.6
2,4-DB	0.2	2,4-DB	6.6
Dalapon	0.1	Dalapon	3.3
Dicamba	0.1	Dicamba	3.3
Dichlorprop	0.2	Dichlorprop	6.6
Dinoseb	0.1	Dinoseb	3.3
MCPA	20	MCPA	660
MCP	20	MCP	660

Table 22.2 SW 8151A LCS Recovery Limits, current at time of SOP revision

Table 22.2 - SW 8151A LCS Recovery Limits, current at time of SOP revision. LCS Recoveries are reviewed and updated semi-annually and are subject to change.									
Analyte - Water Matrix	SPK	Low	High	RPD	Analyte - Soil Matrix	SPK	Low	High	RPD
2,4,5-T	2.5	44	122	30	2,4,5-T	83.3	50	150	30
2,4,5-TP (Silvex)	2.5	49	126	30	2,4,5-TP (Silvex)	83.3	50	150	30
2,4-D	2.5	39	120	30	2,4-D	83.3	40	150	30
2,4-DB	2.5	44	120	30	2,4-DB	83.3	40	150	30
Dalapon	2.5	40	120	30	Dalapon	83.3	30	150	30
Dicamba	2.5	60	120	30	Dicamba	83.3	40	150	30
Dichlorprop	2.5	68	122	30	Dichlorprop	83.3	40	150	30
Dinoseb	2.5	28	115	30	Dinoseb	83.3	40	150	30
MCPA	250	62	144	30	MCPA	8333	40	150	30



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Table 22.2 - SW 8151A LCS Recovery Limits, current at time of SOP revision. LCS Recoveries are reviewed and updated semi-annually and are subject to change.									
Analyte - Water Matrix	SPK	Low	High	RPD	Analyte - Soil Matrix	SPK	Low	High	RPD
MCPP	250	60	133	30	MCPP	8333	40	150	30
DCAA (Surrogate)	5.0	50	130	30	DCAA (Surrogate)	166.7	30	150	30

Table 22.3 Summary of Calibration and QC Procedures

Table 22.3 - Summary of Calibration and QC Procedures for Method SW8151A			
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point initial calibration for all analytes.	Initial calibration prior to sample analysis.	Linear - RSD for all analytes $\leq 20\%$; or Linear - $r > 0.995$; or Non-linear - $r^2 > 0.990$ (6 points must be used).	Correct problem then repeat initial calibration.
Second-source initial calibration verification.	Once per multi-point initial calibration.	All analytes within $\pm 20\%$ of expected value.	Correct problem then repeat initial calibration.
Absolute RT position established for each analyte and surrogate	Set with each ICAL and reset at the beginning of each (12-hr) shift using the CCV.	Position shall be set using the ICAL midpoint standard, or set with the value of the CCV that is run at start of each 12-hr shift.	N/A
CCV RT widow verification for each analyte and surrogate	Each CCV.	All analytes and surrogates in CCV must fall within the RT windows	Correct problem then reanalyze CCV and all samples analyzed since the last acceptable RT verification. If CCV fails RT verification again, redo ICAL and reset RT widow & position.
Continuing Calibration verification.	At the start of each 12-hour shift, at the end of the 12-hr shift or after every 20 samples, whichever first, and at the end of the analysis sequence, or every 10 samples when required by a client QA Plan.	All analytes within $\pm 20\%$ of expected value.	Correct problem, repeat the CCV, if repeat CCV fails, repeat ICAL and reanalyze all samples since last successful CCV.
Method blank.	One per preparation batch.	No analytes detected $> \frac{1}{2}$ MQL.	Correct problem then re-extract and analyze method blank and all samples processed with the



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Table 22.3 - Summary of Calibration and QC Procedures for Method SW8151A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
			contaminated blank.
LCS for all analytes.	One LCS per preparation batch.	QC acceptance criteria, Table 21.2.	Correct problem then re-extract and analyze the LCS and all samples in the affected preparation batch.
MS/MSD for all analytes.	One MS/MSD per every 20 samples.	QC acceptance criteria, Table 21.2., RPD < 30%	Correct problem if determined that failure is unrelated to matrix, then re-extract and analyze the MS/MSD and all samples in the affected preparation batch.
Surrogate (DCAA)	Added to each sample	QC acceptance criteria, Table 21.2	For all QC and field samples, correct problem, re-prep and reanalyze samples with failing surrogates in the associated prep batch, provided sufficient sample is available. If obvious chromatographic interference with the surrogate is present, reanalysis may not be necessary. Refer to Method 8000C, Section 9.6 Requirements and describe in the Laboratory Review Checklist as necessary.
Demonstrate ability to generate acceptable accuracy and precision using four replicate LCS analyses.	Once per analyst.	QC acceptance criteria, Table 21.2.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria.
Second column confirmation of detections.	100% for all positive results.	Same as for initial or primary column analysis, RPD should be <40 %.	Same as for initial or primary column analysis. If RPD >40 %, evaluate chromatography, for co-elutions. Apply P flag to data where RPD > 40%. In general, report lower value when RPD >40 %. Report higher value when RPD <40%.

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AIHA Laboratory Accreditation Programs, LLC

acknowledges that

ALS Laboratory Group, Environmental Division, (Cincinnati)

4388 Glendale-Milford Road, Cincinnati, OH 45242

Laboratory ID: 100921

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005 international standard, *General Requirements for the Competence of Testing and Calibration Laboratories* in the following:

LABORATORY ACCREDITATION PROGRAMS

- | | |
|---|--|
| <input checked="" type="checkbox"/> INDUSTRIAL HYGIENE | Accreditation Expires: November 01, 2018 |
| <input checked="" type="checkbox"/> ENVIRONMENTAL LEAD | Accreditation Expires: November 01, 2018 |
| <input type="checkbox"/> ENVIRONMENTAL MICROBIOLOGY | Accreditation Expires: |
| <input type="checkbox"/> FOOD | Accreditation Expires: |
| <input type="checkbox"/> UNIQUE SCOPES | Accreditation Expires: |

Specific Field(s) of Testing (FoT)/Method(s) within each Accreditation Program for which the above named laboratory maintains accreditation is outlined on the attached **Scope of Accreditation**. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached **Scope of Accreditation**. Please review the AIHA-LAP, LLC website (www.aihaaccreditedlabs.org) for the most current Scope.

William Walsh, CIH
Chairperson, Analytical Accreditation Board

Cheryl O. Morton
Managing Director, AIHA Laboratory Accreditation Programs, LLC



AIHA Laboratory Accreditation Programs, LLC

SCOPE OF ACCREDITATION

ALS Laboratory Group, Environmental Division, (Cincinnati)

4388 Glendale-Milford Road, Cincinnati, OH 45242

Laboratory ID: **100921**

Issue Date: 11/30/2016

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

The EPA recognizes the AIHA-LAP, LLC ELLAP program as meeting the requirements of the National Lead Laboratory Accreditation Program (NLLAP) established under Title X of the Residential Lead-Based Paint Hazard Reduction Act of 1992 and includes paint, soil and dust wipe analysis. Air and composited wipes analyses are not included as part of the NLLAP.

Environmental Lead Laboratory Accreditation Program (ELLAP)

Initial Accreditation Date: 01/31/1995

Field of Testing (FoT)	Technology sub-type/ Detector	Method	Method Description (for internal methods only)
Paint		EPA SW-846 3050B	
		EPA SW-846 6010B	
Soil		EPA SW-846 3050B	
		EPA SW-846 6010B	
Settled Dust by Wipe		EPA SW-846 3050B	
		EPA SW-846 6010B	
Airborne Dust		NIOSH 7300	

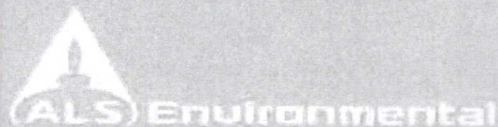
A complete listing of currently accredited Environmental Lead laboratories is available on the AIHA-LAP, LLC website at: <http://www.aihaaccreditedlabs.org>

Not Controlled- please do not copy

ALS Standard Operating Procedure

DOCUMENT TITLE:
REFERENCED METHOD:
SOP ID:
REV. NUMBER:
EFFECTIVE DATE:

DETERMINATION OF TRACE METALS IN SOLUTION BY
INDUCTIVELY COUPLED PLASMA- ATOMIC EMISSION
SPECTROSCOPY BY EPA METHOD 6010B NON-VAP
EPA 6010B
ENV-6010B- NON-VAP
5
SEPTEMBER 1, 2017



STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Determination of Trace Metals in Solution by Inductively Coupled
Plasma-Atomic Emission Spectroscopy by EPA Method 6010B NON-VAP

DOCUMENT CONTROL NUMBER: ENV-6010B-NON-VAP

EFFECTIVE DATE: September 1, 2017

APPROVALS:

LAB DIRECTOR Joseph Rink Date 9/1/17

TECHNICAL MANAGER [Signature] Date 9-1-17

QUALITY ASSURANCE MGR Tracey Earle Date 9-1-17

RECORD OF MINOR REVISIONS:

DATE
9/1/17

LOCATION OF REVISION
Technical review- chg. made to Sec. 8.7.1

QAU APPROVAL
Tracey Earle

RECORD OF MAJOR REVISIONS:

NEW	1	2	3	4	5	6
<u>11/07</u>	<u>10/13</u>	<u>9/14</u>	<u>11/15</u>	<u>8/16</u>	<u>9/17</u>	<u> </u>

**STANDARD OPERATING PROCEDURE
FOR THE DETERMINATION OF TRACE METALS IN
SOLUTION BY INDUCTIVELY COUPLED PLASMA-ATOMIC (ICP-AES)
EMISSION SPECTROSCOPY BY EPA METHOD 6010B**

1.0 SCOPE AND APPLICATION

- 1.1 This method describes multielemental determinations of trace elements in solution by ICP-AES using simultaneous optical systems and axial and/or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photosensitive devices.
- 1.2 Use of this method is restricted to analysts who are knowledgeable in the correction of spectral, chemical and physical interferences described in this method. Instrument operation is restricted to experienced analysts.
- 1.3 This procedure is applicable to the analysis of the following elements.

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc

- 1.4 The list may be modified to include additional elements so long as all standard quality control measures are adhered to. The following are the most routine elements added, although further additions are acceptable as long as all quality control measures are observed.

Boron	Tellurium
Lithium	Tin
Molybdenum	Titanium
Nickel	Tungsten
Phosphorus	Yttrium
Platinum	Zirconium

2.0 SAFETY PRECAUTIONS

- 2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound is treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

- 2.2 Safety glasses, gloves and lab coats must be worn to handle samples, standards and solutions.
- 2.3 ALS Environmental, Cincinnati has a current Radiological License through the Ohio Department of Health. In order to limit exposure to personnel, radiological and/or beryllium containing samples are segregated from non-regulated material at the time of login. All radiological samples are stored in a locked, controlled storage cabinet. Only trained personnel may receive, handle, prepare, store, and dispose of regulated samples as stated in SOPs RAD-003, RAD-004, and RAD-005. Any questions on the handling of radiological samples should be directed to the current RSO.

3.0 SAMPLE HANDLING AND PRESERVATION

- 3.1 Verify the pH of aqueous samples at the time of sample receipt. Acidify aqueous samples to a pH of <2 with HNO₃. Samples for this procedure have a holding time of 6 months after sampling.
- 3.2 Maintain non-aqueous samples at $4 \pm 2^{\circ}\text{C}$ upon receipt by laboratory. Samples for this procedure have a holding time of 6 months after sampling.
- 3.3 Sample digestates have a holding time of 6 months after digestion. However, unless client requests an extended archive time, digestates are only archived for 2 months before disposal.

4.0 DETECTION LIMITS

- 4.1 Reporting limits – The standard reporting limits for routine matrices are listed in LIMS.
- 4.2 The values must be adjusted if effected by the sample matrix or to take into account dilution of the sample.
- 4.3 The values may be adjusted to fulfill project requirements. Lower level reporting limits must be supported by a method detection limit study or reporting limit verification.

5.0 INTERFERENCES

- 5.1 Evaluate interferences for each individual instrument prior to the analysis of samples. Interferences are unique to the instrument and the same wavelength may have different interelement corrections and background point corrections on a different instrument.
 - 5.1.1 Determine the location for off-line background correction by scanning the interference check solution (containing aluminum, calcium, iron and magnesium at a minimum concentration of 100ppm. Additional elements may be added) and comparing the scan to the wavelength of interest. Refer to the instrument manufacturer's instruction for application of the background correction point. The background correction point corrects for background shift and not interelement spectral interference. Background correction must be determined by an experienced analyst.
 - 5.1.2 Determine the interelement correction (IEC) factors by analyzing single source interfering element standards (aluminum, calcium, iron and magnesium at a minimum of 100ppm. Additional elements may be added). Background correction must be applied prior to determination of IECs. Measure the apparent concentration of analyte of interest resulting from the analysis of the interfering

element. Refer to the instrument manufacturer's instruction for application of the interelement correction factor.

- 5.2 Compensate for physical interferences (high dissolved solids, varying viscosity) by using a high solids nebulizer, diluting sample and/or varying of the instrument sample introduction and plasma parameters. All modifications to the instrument must be performed by an experienced analyst.
- 5.3 Matrix match all samples and standards.

6.0 APPARATUS

- 6.1 Inductively coupled argon plasma optical emission spectrometer with autosampler (Thermo Electron Iris and Thermo Scientific iCAP)
- 6.2 Argon gas supply, welding grade or better
- 6.3 Sample uptake tubing
- 6.4 Volumetric pipettes (100-1000 μ L, 5-10mL, 1-10mL, 10-100 μ L)
- 6.5 Volumetric flasks (1L)
- 6.6 Disposable plastic test tubes for autosampler (16x125mm or 13x125mm)

7.0 REAGENTS

- 7.1 Use reagent or trace metal grade chemicals in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
 - 7.1.1 Concentrated Nitric acid (HNO_3), trace metal grade.
 - 7.1.2 Concentrated Hydrochloric acid (HCl), trace metal grade.
- 7.2 ASTM Type II Water [ASTM D1193-77 (1983)]. All references to water in the method refer to ASTM Type II unless otherwise specified.

8.0 CALIBRATIONS

- 8.1 Purchase intermediate standards with all elements of interest at a concentration of 1000mg/L. Standards must be NIST traceable and be provided with an expiration date by the manufacturer. NOTES: Al, Ca, Fe, Pb, Mg, K and Na are required at concentrations of 10000 mg/L to accommodate study ranges. Record all standard preparation information in the working standard (WS) logbook.
- 8.2 Calibration standards: prepare the standard by pipetting the indicated volume of intermediate standard into approximately 750mL of water that has been acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. This list may be modified to accommodate fewer or additional elements.

For calibration standards prepared at 10 ppm

Analyte	Concentration of Intermediate Standard (mg/L)	Volume of Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
¹ Mix A	1000	10.0	1000	10.0
² Mix B	1000	10.0	1000	10.0
³ Mix C	1000	10.0	1000	10.0
Gallium	1000	10.0	1000	10.0
Indium	1000	10.0	1000	10.0
Platinum	1000	10.0	1000	10.0

¹ Mix A: purchased mixed standard containing Al, As, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, P, K, Na

² Mix B: purchased mixed standard containing Sb, Bi, Mo, Si, Te, Sn, Ti, W, Zr

³ Mix C: purchased mixed standard containing Ba, Be, Li, Mn, Ni, Se, Sr, Tl, V, Y, Zn

For calibration standards prepared at 1.0 ppm

Analyte	Concentration of Intermediate Standard (mg/L)	Volume of Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
¹ Mix A	1000	1.0	1000	1.0
² Mix B	1000	1.0	1000	1.0
³ Mix C	1000	1.0	1000	1.0
Silver	1000	1.0	1000	1.0

¹ Mix A: purchased mixed standard containing Al, As, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, P, K, Na

² Mix B: purchased mixed standard containing Sb, Bi, Mo, Si, Te, Sn, Ti, W, Zr

³ Mix C: purchased mixed standard containing Ba, Be, Li, Mn, Ni, Se, Sr, Tl, V, Y, Zn

- 8.3 Initial Calibration Verification (ICV) / Continuing Calibration Verification (CCV): Prepare the standard by pipetting the indicated volume of intermediate standard into approximately 750mL of water that has been acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. This mix will yield a solution containing Al, As, Ba, Cd, Ca, Cr, Fe, Pb, Mg, Pt, K, Se and Na at 5 ppm. The mix yields a solution containing Sb, Be, B, Bi, Co, Cu, Li, Mn Mo, Ni, P, Si, Sr, Te, Tl, Sn, Ti, V, W, Zn and Zr at 0.5 ppm. This list may be modified to accommodate fewer or additional elements.

Analyte	Concentration of Intermediate Standard (mg/L)	Volume of Aliquot (mL)	Final Volume (mL)
¹ Mix A	1000	5.0	1000
² Mix B	1000	0.5	1000
³ Mix C	100	5.0	1000
Silver	1000	0.5	1000
Gallium	1000	5.0	1000
Indium	1000	5.0	1000
Platinum	1000	5.0	1000

¹ Mix A: purchased mixed standard containing Al, As, Cd, Ca, Cr, Fe, Pb, Mg, K, Na, Ba, Se

² Mix B: purchased mixed standard containing Sb, Bi, Mo, Si, Te, Sn, Ti, W, Zr

³ Mix C: purchased mixed standard containing Be, Li, Mn, Ni, Sr, Tl, V, Y, Zn, B, Co, Cu, P

- 8.3.1 The solutions used to prepare the ICV and CCV must come from a different lot than the calibration standards.

- 8.4 Pb ICV for Lead Wipes: Prepare the standard by pipetting 0.8mL of a 1000ppm single source lead intermediate standard into approximately 750mL of water that has been

acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. This concentration of this standard is 0.8ppm.

- 8.5 Zero calibration standard, initial calibration blank (ICB), continuing calibration blank (CCB): Prepare the solution by adding acids to the reagent water to achieve a final concentration of 5% HCl and 5% HNO₃ by volume.
- 8.6 Lowcheck: The Lowcheck must come from a different lot than the calibration standards. Prepare the standard by pipetting the indicated volume of intermediate standard into approximately 750mL of water that has been acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. This mix will yield a solution containing As, Sb, Ba, Be, B, Bi, Cd, Co, Cr, Cu, Li, Pb, Mn, Mo, Ni, P, Si, Se, Sr, Te, Tl, Sn, Ti, V, W, Zn, and Zr at 20ppb. The mix will yield a solution containing Gallium and Indium at 100ppb and Platinum at 100ppb. This list may be modified to accommodate fewer or additional elements.

Element	Concentration of Intermediate Standard (mg/L)	Volume of Aliquot (mL)	Final Volume (mL)	Final Concentration (mg/L)
¹ Mix A	1000	0.02	1000.	0.02
² Mix B	1000	0.02	1000.	0.02
³ Mix C	100	0.2	1000.	0.02
Silver	1000	0.02	1000.	0.02
Gallium	1000	0.1	1000.	0.1
Indium	1000	0.1	1000.	0.1
Platinum	1000	0.1	1000.	0.1

¹ Mix A: purchased mixed standard containing Al, As, Ba, Cd, Ca, Cr, Fe, Pb, Mg, K, Na, Se

² Mix B: purchased mixed standard containing Sb, Bi, Mo, Si, Te, Sn, Ti, W, Zr

³ Mix C: purchased mixed standard containing Be, B, Co, Cu, Li, Mn, Ni, P, Sr, Tl, V, Y, Zn

- 8.7 Lead Lowcheck: Prepare the lowcheck by pipetting 10mL of a 1000ppm single source lead intermediate standard into approximately 750mL of water that has been acidified to match the matrix of the intermediate standard (typically 2% nitric acid). Dilute the standard to a final volume of 1L and mix thoroughly. The concentration of this solution is 100ppm.

8.7.1 Spikes for the lead lowcheck solution are as follows:

- Paints spiked at 0.1 ppm
- Soils/bulks spiked at 0.05 ppm
- Wipes spiked at 0.04 ppm
- Filters spiked at 0.05ppm

- 8.8 Interference Check Solution A: Prepare the standard by pipetting 50mL of the ICSA intermediate standard into approximately 750mL of water that has been acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. The ICSA intermediate standard is purchased at concentrations of 5000ppm for Al, Ca, Mg and 2000ppm for Fe.
- 8.9 Interference Check Solution AB: Prepare the standard by pipetting 50mL of the ICSA intermediate standard and 10mL of the ICSAB intermediate standard into approximately 750mL of water that has been acidified with 50mL of nitric acid and 50mL of hydrochloric acid. Dilute the standard to a final volume of 1L and mix thoroughly. The ICSAB working standard is purchased at concentrations of 50ppm for Ba, Be, Cr, Co, Cu, Mn, V and 100ppm for Cd, Pb, Ni, Ag, Zn.

8.10 The analyte concentrations and acid amounts may be varied to accommodate non routine analyses or to achieve specific project goals.

8.11 All prepared standards expire one year from the date of preparation.

9.0 SAMPLE PREPARATION

9.1 This procedure is for the analysis of previously prepared samples. Refer to the appropriate sample preparation SOP for guidance.

9.1.1 Soil – DCLC SOP “ENV-3050B”

9.1.2 Water – DCLC SOP “ENV-3010A”

9.1.3 Wipes- “ENV-3050B”

10.0 DIAGRAMS OR TABLES

10.1 None

11.0 PROCEDURE

11.1 Fill the rinse reservoir.

11.2 Connect pump winding and insert the sample probe into distilled water.

11.3 Follow the manufacturer’s instructions for instrument start-up and plasma ignition. A default setting is provided by the manufacturer and must not be modified unless instructed by the manufacturer.

11.4 Allow the plasma to equilibrate for one hour. An equilibration time of less than an hour may result in drift.

11.5 If applicable, perform an alignment/profile. This function is both hardware and software dependent. Refer to the manufacturer’s instructions for guidance.

11.6 Standards and samples may be analyzed manually or using an autosampler.

11.6.1 The autosampler is controlled through the instrument software. Refer to the manufacturer’s instruction. The instrument software controls the calibration, QC checks and sample analysis automatically.

11.6.2 Perform manual analysis by inserting the probe into each sample. Acquire the reading using the instrument operating software. Care must be taken to assure sufficient rinse time between sample to prevent carryover.

11.7 Perform the analysis in this order: calibration, ICV, ICB, Lowcheck, ICSA, ICSAB, a maximum of 10 samples, CCV, CCB. Repeat the analysis of a maximum of ten samples followed by a CCV, CCB as necessary. Upon completion of all sample analysis, an ICSA, ICSAB, CCV and CCB must be analyzed.

11.7.1 When analyzing lead wipes, a 0.8ppm Pb ICV must be analyzed prior to the analysis of any lead wipe samples.

12.0 CALCULATIONS

- 12.1 Follow the instrument manufacturer's instructions for data entry of the sample weight and dilution to obtain the desired units. Derive the concentration from a total of 3 exposures per reading. Examples of proper dilution correction and unit conversion are shown below.

For solid samples:

$$\text{Sample Concentration } (\mu\text{g/g}) = \frac{[(\mu\text{g/mL}) \text{ in solution}] * [\text{Final Volume (mL)}]}{[\text{Sample Weight (g)}]}$$

For aqueous samples:

$$\text{Sample Concentration (mg/L)} = \frac{[(\mu\text{g/mL}) \text{ in solution}] * [\text{Final Volume (mL)}] * [1000(\text{mL/L})]}{[\text{Sample Aliquot (mL)}]}$$

For filter and wipe samples:

$$\text{Sample Concentration (ug/sample)} = [(\mu\text{g/mL}) \text{ From Instrument}] * [\text{Final Volume (mL)}]$$

- 12.2 Determination of dry weight fraction.

- 12.2.1 Weigh 5 to 10 grams of sample onto a preweighed aluminum weighing pan. Dry in oven for at least 4 hours at 105°C. Determine the dry weight concentration of the analyte by dividing the results by the dry weight fraction.

$$\% \text{ Dry Weight} = 100 * \frac{\text{Weight dry sample}}{\text{Weight of sample}}$$

- 12.3 Percent recovery calculation:

$$\text{Percent Recovery} = 100 * \frac{\text{Measured Value}}{\text{Target Value}}$$

Matrix Spike Percent Recovery =

$$100 * \frac{\text{Spiked Sample Value} - \text{Unspiked Sample Value}}{\text{Spike Amount}}$$

- 12.4 Precision calculation:

$$\text{Relative Percent difference (RPD)} = 100 * \frac{|V_1 - V_2|}{(V_1 + V_2)/2}$$

where:

V_1, V_2 = found concentrations

13.0 QUALITY ASSURANCE PROVISIONS

13.1 Verify the instrument standardization as follows:

13.1.1 Verify the instrument calibrations using the ICV and ICB. Results obtained from the analysis of the ICV must be within $\pm 10\%$ of the true values for all analytes. If not, terminate the analysis, correct the problem, and, if necessary, re-calibrate the instrument. The results of the ICB must be below the reporting limit. If not, terminate the analysis, correct the problem, and, if necessary, re-calibrate the instrument.

13.1.2 Verify the instrument's capability to produce accurate results at low concentrations. The lowcheck concentration of an analyte should be less than the reporting limit. Should the reporting limit of an analyte be less than the lowcheck concentration, properly dilute the lowcheck to achieve a concentration lower than that of the reporting limit when possible. The results obtained from the analysis of the lowcheck should be within $\pm 20\%$ of the true value for all analytes of interest. If not, the analyst should terminate the analysis, correct the problem, and re-calibrate the instrument.

13.1.3 Verify stability of the calibration every 10 samples and at the end of the analytical run, using the CCB and the CCV standards. The results of the CCV analyses must agree to within $\pm 10\%$ of the true values. If not, terminate the analysis, correct the problem, and, if necessary, re-calibrate the instrument and re-analyze the previous 10 samples. The results of the CCB must be below the reporting limit. If not, terminate the analysis, correct the problem, and, if necessary, re-calibrate the instrument and re-analyze the previous 10 samples.

13.2 Verify the interelement and background correction factors at the beginning and end of the analytical run using the ICSA and ICSAB standards. The results obtained for all analytes of interest in the ICSA/ICSAB must agree to within $\pm 20\%$ of the true value. If not, terminate the analysis, correct the problem, re-calibrate the instrument, and re-analyze all samples since the last valid ICSA/ICSAB analysis.

13.3 Employ a minimum of one reagent blank per sample digestion batch to determine if contamination or memory effects are occurring. A reagent blank is a volume of reagent water acidified with the same amounts of acid as were added to the standards and samples.

The reagent blank control limits and corrective actions are as follows:

Control limits: Less than the reporting limit

Corrective Actions:

- 1) Check for calculation errors, instrument performance
- 2) Re-analyze blank and samples
- 3) Re-prepare and re-analyze samples
- 4) Flag data

13.4 Analyze at least one laboratory control sample (LCS) per sample batch (a maximum of 20 samples of similar matrix).

- 13.4.1 Refer to the specific sample preparation procedure for LCS preparation and concentrations. The ICV solution may be substituted for the aqueous LCS if no preparation is required for the samples.
- 13.4.1.1 Soil – DCLC SOP “ENV-3050B”.
- 13.4.1.2 Water – DCLC SOP “ENV-3010A”.
- 13.4.1.3 Wipes-“ENV-3050B”
- 13.4.2 Compare the recoveries to the laboratory control limits. If there is insufficient data to generate control limits (minimum 20 analyses), the results must be within $\pm 20\%$ of the target value. If any reported analytes fall outside of the control limits for the LCS, the problem must be corrected. The associated samples must be re-analyzed or re-prepared and re-analyzed for the outlying analytes.
- 13.4.3 Prepare and analyze the LCS in duplicate if there is insufficient material present to prepare a matrix spike/matrix spike duplicate. If there is insufficient data to generate control limits (minimum 20 analyses), the results must be within $\pm 20\%$ of the target value. If any reported analytes fall outside of the control limits for the LCS, the problem must be corrected. The associated samples must be re-analyzed or re-prepared and re-analyzed for the outlying analytes. If there is insufficient data to generate control limits (minimum 20 analyses), the results or the RPD must be less than 20%. If the RPD of the LCS/LCSD analysis exceeds the control limit, flag the report indicating the matrix may be non-homogeneous for that analyte.
- 13.5 Analyze one pair of matrix spike samples (MS and MSD) per analytical batch.
- 13.5.1 The MS/MSD is a pair of aliquots taken from one sample, spiked with known amounts of analytes and brought through the entire sample preparation and analysis process. Refer to the specific sample preparation procedure for MS/MSD preparation and concentrations.
- 13.5.2 Compare the recoveries to the established control limits. Recoveries must be within the established control limits, when the sample result does not exceed 4x the spike amount added. If there is insufficient data to generate control limits (minimum 20 analyses), the results must be within $\pm 25\%$ of the target value. If any reported analytes fall outside of the control limits, flag the report indicating a matrix effect may be present. If there is insufficient data to generate control limits (minimum 20 analyses), the results or the RPD must be less than 20%. If the RPD of the MS/MSD analysis exceeds the control limit, flag the report indicating the matrix may be non-homogeneous for that analyte.
- 13.6 Dilute and re-analyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established. Samples that contain interfering elements whose concentration exceeds the linear range must also be diluted to allow for proper interelement correction.
- 13.7 FOR THE ANALYSIS OF NON-ROUTINE NON-SOIL/WATER MATRICES (i.e.: inks, dyes, adhesives, raw materials, etc.) a serial dilution and or post-digestion spike may be performed to aid in the identification of interferences and low level analytes. These are non-routine tests that require an experienced analyst to allow for proper data

interpretation. These non-standard tests are used if this method is applied to an unknown matrix only and are not required for standard soil and water tests.

- 13.7.1 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution must agree within +/- 10% of the original determination. If not, a chemical or physical interference effect will be suspected and flagged as such.
- 13.7.2 A post-digestion matrix spike may be performed for any analytes (exception: Ag) for which the pre-digestion matrix spike recovery did not fall within control limits, and the sample result did not exceed 4x the spike added. An analyte spike added to a portion of a prepared sample, or its dilution, when applicable, is recovered to within 75% to 125% of the true value. The concentration of the spike addition must be within 10 times and a 100 times the detection limit. If the spike is not recovered within the specified limits, a matrix effect will be suspected and flagged as such.
- 13.8 For dust wipe samples, analyze a laboratory control spike sample (LCS) and duplicate (LCSD). Prepare the LCS/LCSD by aliquoting equal amounts of standard solution onto blank sample collection media. Analyze the pair with the frequency of one pair per batch of samples using the control limits established by the laboratory.
- 13.9 An instrument detection limit (IDL) study must be performed semi-annually, or every time the instrument is adjusted in a way which may affect the IDL's, whichever is more frequent. The IDL's are determined by first creating a standard which contains all of the analytes at concentrations between 3x and 5x the instrument manufacturers suggested IDL's. This standard is then analyzed, under normal operating conditions, seven consecutive times per day, on three non-consecutive days. Each analysis must be performed in the same manner as typical analytical samples are measured, including rinsing between analyses with the reagent blank. The standard deviations obtained from the three sets of seven analyses are averaged. The IDL's are obtained by multiplying the average of the three standard deviations by 3.14.
- 13.10 On a semi-annual basis, the linear range of each analyte must be confirmed or every time the instrument is adjusted in a way which may affect the linear range, whichever is more frequent. This is accomplished by analyzing a linear range verification check standard during a routine analytical run. The results must be no less than 90% of the true value. The highest acceptable concentrations are the highest concentrations which can be reported in samples or QC standards. When results are obtained which exceed these values, the sample or QC standard must be diluted and re-analyzed.
- 13.11 A method detection limit (MDL) study must be performed annually, or every time the instrument is adjusted in a way which may affect the MDLs, whichever is more frequent. Refer to the current laboratory procedure for MDLs.
- 13.12 The Quality Control (QC) criteria for all inorganic radiological samples will follow the protocol in Table 8 (Inorganic Analysis by Inductively Coupled Plasma (ICP) Atomic Emission Spectroscopy in Appendix B of the DOD/DOE Consolidated Quality Systems Manual (QSM), Ver. 5, July 2013.

14.0 REPORTING RESULTS

- 14.1 Report results in the units and format specified by the client or contract.

- 14.2 All validated reports must have an electronic or a hand written signature.

15.0 PREVENTIVE MAINTENANCE

- 15.1 Perform preventative maintenance in accordance with the instrument manufacturer's recommendations. Record all service and maintenance performed in the equipment maintenance logbook.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All laboratory waste is accumulated, stored, and disposed in accordance with all federal and state laws and regulations through a licensed waste disposal contractor. Please refer to SOP- SC-003- "Processed Sample Storage and Disposal" for more information.

17.0 REFERENCES

- 17.2 Standard Methods, Method 2540B- "Total Solids Dried at 103-105°C", 1997 editorial revisions 2011.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

EFFECTIVE DATE: September 8, 2017

APPROVALS:

QUALITY ASSURANCE MANAGER *Tracy Gable* Date *9-8-17*

RECORD OF MINOR REVISIONS:

QAU APPROVAL
Tracey Earle

RECORD OF MAJOR REVISIONS:

NEW	1	2	3	4	5	6
<u>1/93</u>	<u>1/97</u>	<u>5/01</u>	<u>2/08</u>	<u>4/09</u>	<u>1/15</u>	<u>7/16</u>
7	8	9	10	11	12	13
<u>9/17</u>						

STANDARD OPERATING PROCEDURE
PREVENTIVE MAINTENANCE
FOR ANALYTICAL INSTRUMENTATION

1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure (SOP) provides procedures for preventive maintenance of specific laboratory instrumentation. Calibration and maintenance of pipettes, balances, and refrigerator thermometers are described in separate SOPs.
- 1.2 Table 1 of this SOP, which is the Preventive Maintenance Schedule and Parts List, provides the frequency requirements for each maintenance activity.

2.0 DEFINITIONS

- 2.1 **Preventive Maintenance** - Any repair activity or procedure performed on instrument hardware or electronics for the purpose of assuring the continued quality performance of the instrument.

3.0 RESPONSIBILITIES

- 3.1 Each analyst/technician is responsible for maintenance of equipment immediately prior to analytical testing. Activities and data related to instrument maintenance shall be documented in the Instrument Maintenance Logbook. The analyst/technician performing the maintenance is responsible for such documentation.
- 3.2 Section Managers are responsible for (1) assurance of maintenance in accordance with Table 1 of this SOP; (2) notification of the manufacturer's or vendor's service engineer or representative when outside service is required; (3) assurance that maintenance is performed in accordance with manufacturer's specifications; (4) the maintenance of an adequate supply of critical spare parts to minimize instrument downtime; and (5) assurance that the maintenance activities are documented in compliance with laboratory standard operating procedures.

4.0 GENERAL PROCEDURES

- 4.1 Maintenance schedules for analytical instruments should be those recommended by the instrument manufacturer, unless experience dictates otherwise. Frequency requirements for preventive maintenance procedures are specified in Table 1 of this SOP. The frequency of preventative maintenance may be altered according to analyst experience and judgement. Table 1 is the minimum suggested Preventive Maintenance Schedule and Parts List for specific laboratory instrumentation.
- 4.2 If an instrument does not meet calibration requirements of an analytical method or is found to be performing unsatisfactorily with respect to method QC criteria, it will not be

utilized for sample analysis until repairs have been completed. The instrument will be isolated to prevent its use or clearly labeled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly.

- 4.3 An instrument identification number will be assigned to each instrument by the appropriate Section Manager and will be clearly posted on each instrument. A complete list of pertinent equipment and instruments will be maintained.
- 4.4 Maintenance records will be kept for all maintenance performed, including external servicing when required. Documentation will be recorded in the Instrument Maintenance Logbook for the specific instrument by the individual responsible for the maintenance procedure. Laboratory personnel may document external servicing, when necessary.
- 4.5 ALS measuring instruments (ex. Balances) are never used outside the laboratory. However, if a measuring instrument is taken out of service for repair or any time a measuring instrument is handled, transported, or stored, the manufacturers' instructions will be observed. All instruments must then be recalibrated by the analyst before use.
- 4.6 Critical spare parts are defined in Table 1. Each Section Manager will maintain an adequate supply of critical spare parts for pertinent instrumentation to minimize instrument downtime.

5.0 PREVENTIVE MAINTENANCE PROCEDURES FOR GC/MS INSTRUMENTATION

5.1 Routine Preventive Maintenance for Hewlett-Packard 5972 and 5973 GC/MSD systems - Source Cleaning - Injection Port Maintenance

5.1.1 *Removing the Analyzer*

- 5.1.1.1 Shutdown and venting of the MSD system must be performed prior to installation or replacement of a capillary column and prior to many maintenance tasks.
- 5.1.1.2 On the data system, select the vacuum control program.
- 5.1.1.3 Select the vent option. This initiates the venting program.
- 5.1.1.4 The data system will display a message instructing the analyst to turn off the GC/MSD heater and the GC oven.
- 5.1.1.5 After the heated zones have been turned off, the data system will display the diffusion pump status, MS (analyzer) temperature, and the foreline pressure. It will also provide an estimate of the time that it will take the diffusion pump and analyzer to cool.
- 5.1.1.6 When the time has elapsed or when the program prompts you to do so, switch off the on/off switch on the back of the MSD.

- 5.1.1.7 The system has been successfully vented and maintenance can now be performed.
- 5.1.1.8 Disconnect the MSD power cord and carefully slide the MSD away from the GC.
- 5.1.1.9 Remove the screws that hold the MSD top cover in place and remove the top cover.
- 5.1.1.10 Remove the clamp from the KF25 fitting which connects the GC/MSD interface to the MSD.
- 5.1.1.11 Carefully pull the GC/MSD interface out of the vacuum manifold and set it on a clean, lint-free cloth, then cover it with another clean cloth. Do not touch portions of the interface which are normally inside the vacuum manifold.
- 5.1.1.12 Disconnect the electrical connections between the top board and the main board.
 - 5.1.1.12.1 Unplug the ribbon cable from the top board.
 - 5.1.1.12.2 Unplug the filament supply cable from the top board.
 - 5.1.1.12.3 Unscrew the detector output cable from the top board.
- 5.1.1.13 If shipping clamps are still in place (they should not be used during normal operation), loosen the rear clamp and remove the front clamp.
- 5.1.1.14 Lift the analyzer/top plate/top board out of the vacuum manifold and place it upside down on a clean work surface.

5.1.2 *Source Disassembly and Cleaning*

- 5.1.2.1 Examine the wires between the nine-pin feedthrough and the pins of the ion source. Make notes about which wires connect to which pins on the ion source to assist with reconnection later.
- 5.1.2.2 Disconnect the wires from the pins on the ion source.
- 5.1.2.3 Unscrew and remove the GC/MS interface socket.
- 5.1.2.4 Remove the two small screws that hold the ion source into the radiator.
- 5.1.2.5 Pull the ion source away from the housing being careful not to damage the filaments.
- 5.1.2.6 Place the ion source on a clean, lint-free cloth.
- 5.1.2.7 Remove the two filament assemblies being careful not to bend the filaments.

- 5.1.2.8 Remove the two screws that hold the repeller in place and remove the repeller.
- 5.1.2.9 Remove the set screw that holds the lens stack in place.
- 5.1.2.10 From the repeller end of the ion source, push out the lens stack (entrance lens, ion focus lens, and insulator), drawout cylinder and drawout plate. Do not apply force to the electrical pins on the entrance lens or ion focus lens.
- 5.1.2.11 Almost all of the metal parts in the ion source are stainless steel and are relatively easy to clean with solvents and mild abrasives. The manufacturer recommends a three step process for cleaning stainless steel parts: abrasive cleaning, ultrasonic cleaning in a series of solvents, and drying.
- 5.1.2.11.1 Abrasive paper or a slurry of alumina powder and methanol can be used to clean steel parts. Generally the slurry is preferred because it is less abrasive and a swab with the slurry can reach corners that may be difficult to clean with abrasive paper.
- 5.1.2.11.2 Rinse all residue away from the abrasive cleaning using reagent grade methanol prior to ultrasonic cleaning.
- 5.1.2.11.3 All steel parts, whether abrasively cleaned or not, should be ultrasonically cleaned for at least 15 minutes in each of the following solvents: methylene chloride, acetone, and methanol.
- 5.1.2.11.4 Drying can be accomplished by baking the cleaned parts in the GC oven at 50°C for 30 minutes.
- 5.1.2.12 Some parts of the MSD are not stainless steel and require special or no cleaning.
- 5.1.2.12.1 The filament assemblies should not be cleaned at all. Attempting to clean the filaments could easily result in damage to the assemblies.
- 5.1.2.12.2 The repeller is mostly stainless steel, but has a vespel seal. The stainless portion may be cleaned as in the 5.1.2.11 above, but the vespel seal should not be abrasively cleaned. The seal may safely be ultrasonically cleaned.
- 5.1.2.12.3 The lens insulators are also vespel and should not be cleaned at all. Abrasive cleaning will damage the insulator and ultrasonic cleaning will cause the insulators to swell.

5.1.3 *Reassembly of the source*

- 5.1.3.1 Always wear clean gloves and have a clean work surface to avoid recontamination of cleaned parts.
- 5.1.3.2 Slide the drawout plate and then the drawout cylinder into the ion source body.
- 5.1.3.3 Bring together the entrance lens, ion focus lens, and the two lens insulators. Together these make up the lens stack.
- 5.1.3.4 Slide the lens stack into the source body.
- 5.1.3.5 Reinstall the set screw that holds the lens stack in place.
- 5.1.3.6 Reinstall the repeller on the source body.
- 5.1.3.7 Reinstall the filament assemblies. Make sure that the filaments are still centered over the small holes in the source body.

5.1.4 *Reinstalling the ion source and the analyzer*

- 5.1.4.1 Reinstall the ion source in the radiator. Be careful not to damage the filaments.
- 5.1.4.2 Reinstall the GC/MSD interface socket.
- 5.1.4.3 Reconnect the wires from the feedthrough to the appropriate pins on the ion source. Refer back to the notes that were taken during disassembly for correct configuration.
- 5.1.4.4 Reinstall the analyzer in the MSD.
- 5.1.4.5 Lift the analyzer (with the top plate and top board) from the work surface and invert the assembly so that the analyzer is hanging below the top plate. Do not touch any part of the analyzer or the analyzer side of the top plate.
- 5.1.4.6 Lower the analyzer into position in the vacuum manifold. Make sure that the top plate is resting on the top plate seal.
- 5.1.4.7 Reconnect the ribbon cable, filament supply cable, and detector output cable.
- 5.1.4.8 Reinstall the KF25 O-ring assembly on the GC/MSD interface KF flange. If the O-ring is worn or damaged, replace it.
- 5.1.4.9 Gently insert the GC/MSD interface back into the vacuum manifold. Make sure that the tip of the GC/MSD interface extends into the ion

source and that the interface sits properly in the interface socket on the radiator.

5.1.4.10 Slide the MSD back into position next to your gas chromatograph.

5.1.4.11 Reinstall the transfer line (capillary column).

5.1.4.12 Reinstall the MSD cover.

5.1.4.13 Reconnect the MSD power cord.

5.1.4.14 Turn the MSD power on. The foreline pump should begin with a gurgling sound that should become quieter and stop within 1 minute.

5.1.4.14.1 If the gurgling does not stop, a major air leak is indicated.

5.1.4.14.2 Switch the MSD off and locate and correct the source of the leak.

5.1.4.14.3 Turn the MSD power back on and listen for the gurgling noise to stop.

5.1.4.15 Pump down the MSD using the pumpdown option of the data systems vacuum control option. This again display the diffusion pump status, MS (analyzer) temperature, and the foreline pressure along with a time estimate of how long it will take the diffusion pump to reach it's normal operating temperature.

5.1.4.16 When the diffusion pump reaches it's normal operating temperature, you will receive a prompt to turn on the GC/MSD interface heater and the GC oven heater.

5.1.4.17 Wait at least 4 hours for the MSD to reach thermal equilibrium, then perform an autotune or tune the MSD manually.

5.1.5 *Injection port Maintenance*

5.1.5.1 Cleaning the injection port area is the first corrective action to be tried when initial or continuing calibration criteria can not be met, or when peak shape, particularly of phenols, indicates a chromatography problem.

5.1.5.2 Instrument preparation - Prior to initiating the injection port cleaning, the instrument must be vented and the GC oven cooled to room temperature.

5.1.5.2.1 Turn the injector "A" to off using the GC oven control panel. Allow the injector to cool to room temperature before performing any procedures.

5.1.5.3 Removal of injection port components

- 5.1.5.3.1 Using the injection port wrench remove the top nut of the injector.
- 5.1.5.3.2 Inspect the injector septa for cracks and large holes. Replace the septa (Supelco Thermogreen LB-1 or LB-2, 12.5 mm diameter), if necessary, and reinstall the septa nut. Install the nut to slightly more than finger tight. Overtightening will cause broken injector syringes and will prohibit successful sample introduction.
- 5.1.5.3.3 With the injection port wrench, loosen the split/splitless weldment assembly under the gas lines serving the injection port and remove the deactivated glass liner and O-ring. Inspect the O-ring and replace if worn or damaged. The deactivated liner should be replaced when analyte responses have deteriorated and no longer meet method specifications.
- 5.1.5.3.4 Insert a new injection liner and replace the O-ring. Replace the split/splitless weldment assembly and tighten.
- 5.1.5.3.5 Using a 5/16" wrench, remove the small nut at the bottom of the injection port (inside the oven), and remove the column. If the ferrule is stuck tight, grasp it as gently as possible with a small pair of needle-nosed pliers, and rotate it to free the ferrule and the column.
- 5.1.5.3.6 Remove the nut at the bottom of the injection port, inside the oven, and inspect the gold seal. If the seal is dirty or damaged, replace it and reinstall the bottom of the injection port.
- 5.1.5.3.7 Score the column and break at approximately one foot from the existing nut and ferrule. Discard the short column portion that has been severed. Place a new injector nut then new ferrule (Supeltex M2-A 0.5 mm i.d. graphite-vessel or equivalent) over the column and slide down and out of the way. Examine the column, and make a clean break point below any discoloration of the column. It is important to make the final break after the ferrule is on the column to avoid the possibility of either chipping the column end or contaminating the opening with graphite shavings. Measure 105 mm from the end of the column and, with the ferrule between the end of the column and the mark, mark the column with correction fluid.
- 5.1.5.3.8 Insert the column into the bottom of the port, taking care to not chip the end. Slide the column up into the port so that the ferrule backs against the mark made in 5.1.9. Holding the column in place, slide the 5/16" nut up over the ferrule and quickly thread the nut onto the port and tighten to finger-tight. Check to make sure that the mark is flush with

the bottom of the nut. When the adjustment is correct, tighten the nut.

- 5.1.5.3.9 Close the oven door, reset the injector temperature, and proceed to analysis.

5.1.6 *Pump Maintenance*

NOTE: Avoid contact with the pump oil which possibly contains trace amounts of toxic and carcinogenic materials.

- 5.1.6.1 Pump maintenance should be performed in conjunction with analyzer cleaning and service and only when the analyzer is properly vented.
- 5.1.6.2 Unplug the pump.
- 5.1.6.3 Disconnect the KF25 clamp from the bottom of the pellet trap.
- 5.1.6.4 Place the pump on the edge of a stable surface and acquire a reservoir for the used oil.
- 5.1.6.5 Remove the oil fill plug and hold the oil reservoir under the drain plug.
- 5.1.6.6 Slowly loosen the drain plug until oil starts to leak from it.
- 5.1.6.7 Allow the used oil to collect in the reservoir until it has stopped leaking from the pump.
- 5.1.6.8 Reinstall the drain plug.
- 5.1.6.9 Pour approximately 300 mL of oil into the pump fill hole.
- 5.1.6.10 Watch the fluid level window. Add oil until the fluid level is near, but not above, the upper mark next to the fill window.
- 5.1.6.11 Reinstall the fill plug.
- 5.1.6.12 Replace the pump to its position near the instrument and reattach the KF25 clamp.
- 5.1.6.13 Plug in the pump to the power supply.
- 5.1.6.14 Pump maintenance is completed.
- 5.1.7 Check all fans in the instrument. Clean the dust from their screens and verify that they are turning.
- 5.1.7.1 Any fans found to be nonfunctioning must be repaired or replaced. Contact the Section Manager or designee.

- 5.1.8 *Documentation* - All of the procedures performed in the PM must be documented in the Instrument Maintenance Logbook. Any abnormalities detected in the process should be recorded.

5.2 **Other GC/MS Instrumentation**

- 5.2.1 Additional GC/MS instrumentation other than specified above may be utilized for sample analyses. Preventive maintenance for these instruments must be performed at the frequency specified in Table 1 and in accordance with manufacturer's specifications.
- 5.2.2 Section Managers will provide procedures from the instrument operation, maintenance or service manuals for each maintenance activity.

6.0 **PREVENTIVE MAINTENANCE PROCEDURES FOR GAS CHROMATOGRAPHY INSTRUMENTATION**

- 6.1 **Septum replacement** - Remove the septum nut. Remove the old septum and replace with a new one. Replace the septum nut, hand tighten, and then tighten 1/4 turn more. Do not overtighten the septum nut.

WARNING: The injection port and septum nut can be very hot.

- 6.2 **Injection port liner replacement** - Remove the split/splitless weldment assembly, exposing the top of the injection port liner. Lift the liner out of the injection port. Clean or replace with a new liner packed loosely with a small plug of deactivated glass wool. Replace the O-ring at the top of the liner. Replace the weldment assembly, making sure the O-ring seals are around the insert.
- 6.3 **Detector maintenance** - Refer to the manufacturer maintenance manual for specific maintenance procedures for each type of detector. Routine maintenance can include: cleaning and/or replacing jets and collectors, replacing O-rings, seals, lamps, and reaction tubes, and removal of the detector for service performed by the manufacturer.

7.0 **PROCEDURES FOR SERVICE CONTRACTS OR ALTERNATE SERVICE ARRANGEMENTS**

- 7.1 Instrumentation is maintained by either a manufacturer service contracts or the analyst. It is the responsibility of the analyst to perform all routine maintenance and repairs. For instruments that are not under service contract, after it has been determined by the appropriate Section Manager that an instrument problem is beyond the scope of the analyst, service from the instrument vendor may be solicited.
- 7.1.2 For instruments under service contract - To minimize downtime, the vendor providing the service will be notified by section management as soon as possible when a problem arises.

8.0 DEIONIZED WATER SUPPLY MAINTENANCE

- 8.1 The QA Manager, or appointed designee, is responsible for the monitoring of the deionized water (Type II) system and arranging for maintenance when necessary.
- 8.2 Maintenance for the system is performed in-house and is recorded in a logbook in the Spectroscopy lab.
- 8.3 A logbook is kept in the QA Office to monitor the conductivity of the laboratory high purity water system on a regular basis.

9.0 MICROSCOPY PREVENTATIVE MAINTENANCE

- 9.1 The Microscopy section management and analysts are responsible for monitoring and arranging for the required annual service on the microscopes.
- 9.2 Service will be provided by qualified technicians from an acceptable service provider.
- 9.3 Documentation of all service, including annual service, is maintained in the microscope's maintenance logbooks.

10.0 RADIATION SAFETY EQUIPMENT

- 10.1 It is the responsibility of the Radiation Safety Officer (RSO), or his designee, to ensure that preventative maintenance and equipment repair is provided by an acceptable service provider.
- 10.2 Records for maintenance on radiation surveying equipment are maintained by the RSO.
- 10.3 Radiation surveying equipment that is not performing accurately will be removed from service by the RSO until the equipment is returned to acceptable working condition.

11.0 CORRECTIVE ACTION PROCEDURES

- 11.1 All nonconformances and corrective actions shall follow laboratory standard operating procedure.

12.0 DOCUMENTATION

- 12.1 An ALS Property number is assigned to each instrument by the QA Instrument Maintenance Logbooks are assigned to each instrument for recording all maintenance activities associated with the instrument including daily activities. One or more pages in the front of the logbook are designated for signatures of analysts performing maintenance. The following pertinent information will identify the logbook: instrument description, manufacturer, model number, and ALS identification number. Documentation in the Instrument Maintenance Logbook will be in accordance with the procedures for general laboratory documentation requirements.

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- 12.2 A Logbook is kept in the QA Office to record the conductivity on a regular basis of the laboratory high purity water system (See SOP QA-011- Supporting Equipment Calibrations and Verifications). The procedures for general laboratory documentation requirements will be followed.

13.0 ATTACHMENTS

- 13.1 Table 1: Preventative Maintenance Schedule and Parts List.